

US Application 09/578,420

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1999DE121

The invention relates to a process for the preparation of (metal) salts of alkylphosphonous acids, and to the use of the (metal) salts of alkylphosphonous acids prepared by this process.

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Organic phosphorus compounds are increasing in industrial importance. They are employed principally for the preparation of herbicides or as herbicides themselves, as extractants and as flame retardants. The

10 starting materials used are preferably PH_3 and phosphorus halides, which must themselves in turn be prepared from yellow phosphorus.

The reaction of red phosphorus in the superbasic medium
15 dimethyl sulfoxide/potassium hydroxide (DMSO/KOH) with acetylenes or olefins as alkylating agent (Trofimov et al., Phosphorus, Sulfur and Silicon 55, 271, 1991) preferentially gives triorganylphosphines and triorganylphosphine oxides.

20

The alkylation of red phosphorus using acrylonitrile under the abovementioned conditions with ultrasound treatment of the reaction mixture gives predominantly secondary phosphine oxide. If 1,1-diphenylethylene is
25 employed, tert-phosphine oxide (30%), sec-phosphine

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oxide (10%) and phosphinic acid (35%) are obtained [D. Semenzin et al., Tetrahedron Letters 35, 3297, 1994].

5 It has also been attempted (Trofimov et al., Main Group Chem. News 4, 18, 1996, Phosphorus, Sulfur and Silicon, 109/110, 601, 1996) to react elemental phosphorus in its red modification with alkyl halides in the presence of potassium hydroxide, water, dioxane and a phase-
10 transfer catalyst. The main product found comprised tert-phosphine oxides (up to 75% in the case of benzyl bromide, about 60 - 65% in the case of butyl bromide). As byproducts, sec-phosphine oxides and phosphinic acid esters are obtained with 19% and 6% respectively, but
15 the former only in the presence of zinc powder as reducing agent.

However, the abovementioned processes have the disadvantage that first of all the red phosphorus or
20 organophosphorus intermediates have to be prepared. The processes are technically very complex and consequently also not economical, and the products obtained often have to be purified subsequently at great effort. In particular, the specific preparation of certain
25 compounds in high yield is frequently particularly difficult.

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In addition, a number of starting materials, such as halogen-containing phosphorus compounds or phosphines, are unsuitable from the very beginning for a large-scale and economical process owing to their environmental toxicity.

There is therefore a demand for a process for the preparation of (metal) salts of alkylphosphonous acids which can be carried out in a simple manner and in which uniform products are obtained in high yield. A process of this type should also be clearly superior to those known hitherto in economic and environmental terms.

The invention therefore has the object of providing a process for the preparation of (metal) salts of alkylphosphonous acids which avoids the abovementioned disadvantages and with which the desired end products can be prepared without difficulty.

This object is achieved by a process of the type described at the outset, which comprises reacting elemental yellow phosphorus with alkyl halides in the presence of carbonates, bicarbonates, amides,

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alkoxides, amine bases and/or solid hydroxides as
bases.

The reaction is preferably carried out in a two-phase
5 system comprising at least one base and an organic
solvent.

The alkyl halides employed are preferably methyl
chloride or methyl bromide.

10

The organic solvents employed are preferably straight-
~~chain or branched alkanes,~~ alkyl-substituted aromatic
solvents, water-immiscible or only partially water-
miscible alcohols or ethers, alone or in combination
15 with one another.

The organic solvent employed is particularly preferably
toluene, alone or in combination with alcohols.

20 The reaction is preferably carried out in the presence
of a phase-transfer catalyst.

The phase-transfer catalyst is preferably a tetraalkyl-
phosphonium halide, triphenylalkylphosphonium halide or
25 tetraorganylammonium halide.

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The temperature in the reaction is preferably from -20 to +60°C.

The temperature is particularly preferably from 0 to 5 30°C.

The reaction is preferably carried out under a pressure of from 0 to 10 bar.

10 The process according to the invention is preferably carried out by suspending or dissolving the yellow phosphorus in a solvent or solvent mixture and then reacting it with an alkyl halide and at least one base.

15 The yellow phosphorus and the alkyl halide are preferably reacted with one another in a molar ratio of from 1:1 to 1:3, where the molar ratio of yellow phosphorus to base is from 1:1 to 1:5.

20 The two-phase system obtained after the reaction is preferably separated and processed further as an aqueous phase.

The invention also relates to the use of the (metal)
25 salts of alkylphosphonous acids prepared in accordance

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with the invention as precursors for chemical syntheses.

The invention also relates to the use of the (metal)
5 salts of alkylphosphonous acids prepared in accordance
with the invention for the preparation of organo-
phosphorus compounds.

The invention also relates to the use of the (metal)
10 salts of alkylphosphonous acids prepared in accordance
with the invention as flame retardants or for the
preparation of flame retardants.

The invention relates in particular to the use of the
15 (metal) salts of alkylphosphonous acids prepared in
accordance with the invention for the preparation of
flame retardants for thermoplastic polymers, such as
polyethylene terephthalate, polybutylene terephthalate
or polyamide.

20

The invention relates in particular to the use of the
(metal) salts of alkylphosphonous acids prepared in
accordance with the invention for the preparation of
flame retardants for thermosetting resins, such as
25 unsaturated polyester resins, epoxy resins, poly-
urethanes or acrylates.

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Surprisingly, it has been found that elemental yellow phosphorus can be reacted with alkylating agents in a two-phase system (organic solvent/base) and, if
5 desired, in the presence of a (phase-transfer) catalyst under extremely mild conditions by the process according to the invention to give the (metal) salts of the corresponding alkylphosphonous acid $RP(:O)HOH$.

10 In addition, small amounts of dialkylphosphinic acids, trialkylphosphine oxide $R_3P(:O)$, dialkylphosphine oxide and unidentified phosphorus compounds may be formed; these can be removed from the product mixture in the usual manner. A further byproduct formed is hydrogen,
15 which can easily be separated off from the reaction mixture. The abovementioned dialkylphosphinic acids can be separated off from the reaction mixture and employed or further processed elsewhere.

20 Surprisingly, the process according to the invention gives neither phosphine (PH_3) nor alkylphosphines (RPH_2 , R_2PH) in significant amounts. Through the choice of suitable reaction conditions - such as the addition of small amounts of alcohols to the organic phase - the
25 formation of all unidentified phosphorus-containing byproducts can be minimized to a surprisingly low

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content of a few mol% of the yellow phosphorus employed, in favor of the main product, the (metal) salts of alkylphosphonous acid.

5 The process according to the invention can be carried out, for example, by introducing the solvent together with the phase-transfer catalyst and warming the mixture to above the melting point of the yellow phosphorus, then adding the elemental (yellow) phosphorus, cooling the mixture to temperatures of, for
10 example, from -10 to +30°C with vigorous stirring, and subsequently adding the alkyl halide.

The reaction is initiated by addition of the base. When
15 the reaction is complete, the reaction mixture can be diluted, for example with water, and the readily volatile components (H_2 , PH_3 , RPH_2 , R_2PH and excess alkyl halide, etc.) are subsequently removed.

20 This process gives a base-containing/organic two-phase system whose phases are separated. The contents from the phases are determined analytically.

The base-containing phase can be worked up by the known
25 methods of the prior art in order to obtain the pure

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acids, such as, for example, alkylphosphonous acid (for example by ion exchange or distillation).

The reactants can also be combined in a different sequence, for example by introducing them continuously into a reactor (pressure tube, pressure reactor or cascade) in the above-defined molar ratio and removing them from the reactor again after a residence time of from 0.5 to 2 hours. The organic phase obtained after the phase separation, which still contains the majority of any phase-transfer catalyst employed, is advantageously recycled.

Example 1: Methylphosphonous acid

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A solution of 26.1 g (0.05 mol) of tributylhexadecylphosphonium bromide in 1000 ml of toluene was introduced into a 5 l stainless-steel pressure reactor and pre-heated to 60°C. 31 g (1 mol) of yellow phosphorus were added, the mixture was cooled to -10°C with vigorous stirring, and 202 g (4 mol) of methyl chloride were then metered in. The mixture was then warmed to 20°C, and 500 g of 50% aqueous potassium carbonate solution were metered in over the course of 8 hours, during which the temperature was held at 20°C. Over the course of a further hour, 300 g of water were

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added, the mixture was then stirred for a further two hours, and the reactor was subsequently decompressed via combustion. Two homogeneous liquid phases were obtained, which were separated and analyzed.

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The aqueous phase (weight: 900 g) contained 52.7 mol% of methylphosphonous acid, 18.9 mol% of phosphorous acid, 22.7 mol% of hypophosphorous acid, 2.8 mol% of dimethylphosphinic acid in the form of their potassium salts and 2 mol% of dimethyldiphosphine.

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The acids can be obtained from the salts prepared in Example 1 in a known manner, for example by ion exchange.

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Patent claims

1. A process for the preparation of (metal) salts of alkylphosphonous acids, which comprises reacting
5 elemental yellow phosphorus with alkyl halides in the presence of carbonates, bicarbonates, amides, alkoxides, amine bases and/or solid hydroxides as bases.
- 10 2. A process as claimed in claim 1, wherein the reaction is carried out in a two-phase system comprising at least one base and an organic solvent.
- 15 3. A process as claimed in claim 1 or 2, wherein the alkyl halides employed are methyl chloride or methyl bromide.
- 20 4. A process as claimed in one or more of claims 1 to 3, wherein the organic solvents employed are straight-chain or branched alkanes, alkyl-substituted aromatic solvents, water-immiscible or only partially water-miscible alcohols or ethers, alone or in combination with one another.

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5. A process as claimed in one or more of claims 1 to 4, wherein the organic solvent employed is toluene, alone or in combination with alcohols.
- 5 6. A process as claimed in one or more of claims 1 to 5, wherein the reaction is carried out in the presence of a phase-transfer catalyst.
- 10 7. A process as claimed in claim 6, wherein the phase-transfer catalyst is a tetraalkylphosphonium halide, triphenylalkylphosphonium halide or tetra-organylammonium halide.
- 15 8. A process as claimed in one or more of claims 1 to 7, wherein the temperature during the reaction is from -20 to +60°C.
- 20 9. A process as claimed in one or more of claims 1 to 8, wherein the temperature is from 0 to 30°C.
- 10 10. A process as claimed in one or more of claims 1 to 9, wherein the reaction is carried out at a pressure of from 0 to 10 bar.
- 25 11. A process as claimed in one or more of claims 1 to 10, wherein the yellow phosphorus is suspended or

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dissolved in a solvent or solvent mixture and then reacted with an alkyl halide and at least one base.

5 12. A process as claimed in one or more of claims 1 to 11, wherein the yellow phosphorus and the alkyl halide are reacted in a molar ratio of from 1:1 to 1:3, where the molar ratio of yellow phosphorus to base is from 1:1 to 1:5.

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13. A process as claimed in one or more of claims 1 to 12, wherein the two-phase system obtained after the reaction is separated and further processed.

15 14. The use of a (metal) salt of an alkylphosphonous acid prepared by a process as claimed in claims 1 to 13 as a precursor for chemical syntheses.

15. The use of a (metal) salt of an alkylphosphonous acid prepared by a process as claimed in claims 1 to 13 for the preparation of organophosphorus compounds.

20 16. The use of a (metal) salt of an alkylphosphonous acid prepared by a process as claimed in claims 1 to 13 for the preparation of organophosphorus compounds.

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to 13 as a flame retardant or for the preparation of flame retardants.

17. The use of a (metal) salt of an alkylphosphonous acid prepared by a process as claimed in claims 1 to 13 for the preparation of flame retardants for thermoplastic polymers, such as polyethylene terephthalate, polybutylene terephthalate or polyamide.

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18. The use of a (metal) salt of an alkylphosphonous acid prepared by a process as claimed in claims 1 to 13 for the preparation of flame retardants for thermosetting resins, such as unsaturated polyester resins, epoxy resins, polyurethanes or acrylates.

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Process for the preparation of (metal) salts of
alkylphosphonous acids II

Abstract

The invention relates to a process for the preparation of (metal) salts of alkylphosphonous acids, which comprises reacting elemental yellow phosphorus with alkyl halides in the presence of carbonates, bicarbonates, amides, alkoxides, amine bases and/or solid hydroxides as bases.

The invention also relates to the use of the (metal) salts of alkylphosphonous acids prepared by this process.

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The invention relates to a process for the preparation of (metal) salts of alkylphosphonous acids, and to the use of the (metal) salts of alkylphosphonous acids prepared by this process.

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Organic phosphorus compounds are increasing in industrial importance. They are employed principally for the preparation of herbicides or as herbicides themselves, as extractants and as flame retardants. The starting materials used are preferably PH_3 and phosphorus halides, which must themselves in turn be prepared from yellow phosphorus.

Hitherto, only a few processes were known with which organic phosphorus compounds having more than one phosphorus-carbon bond can be prepared from simple starting materials.

The reaction of red phosphorus in the superbasic medium dimethyl sulfoxide/potassium hydroxide (DMSO/KOH) with acetylenes or olefins as alkylating agent (Trofimov et al., Phosphorus, Sulfur and Silicon 55, 271, 1991) preferentially gives triorganylphosphines and triorganylphosphine oxides.

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The alkylation of red phosphorus using acrylonitrile under the abovementioned conditions with ultrasound treatment of the reaction mixture gives predominantly secondary phosphine oxide. If 1,1-diphenylethylene is employed, tert-phosphine oxide (30%), sec-phosphine oxide (10%) and phosphinic acid (35%) are obtained [D. Semenzin et al., Tetrahedron Letters 35, 3297, 1994].

- 10 It has also been attempted (Trofimov et al., Main Group Chem. News 4, 18, 1996, Phosphorus, Sulfur and Silicon, 109/110, 601, 1996) to react elemental phosphorus in its red modification with alkyl halides in the presence of potassium hydroxide, water, dioxane and a phase-transfer catalyst. The main product found comprised
- 15 tert-phosphine oxides (up to 75% in the case of benzyl bromide, about 60 - 65% in the case of butyl bromide). As byproducts, sec-phosphine oxides and phosphinic acid esters are obtained with 19% and 6% respectively, but
- 20 the former only in the presence of zinc powder as reducing agent.

However, the abovementioned processes have the disadvantage that first of all the red phosphorus or

25 organophosphorus intermediates have to be prepared. The processes are technically very complex and consequently

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also not economical, and the products obtained often have to be purified subsequently at great effort. In particular, the specific preparation of certain compounds in high yield is frequently particularly
5 difficult.

In addition, a number of starting materials, such as halogen-containing phosphorus compounds or phosphines, are unsuitable from the very beginning for a large-
10 scale and economical process owing to their environmental toxicity.

There is therefore a demand for a process for the preparation of (metal) salts of alkylphosphonous acids
15 which can be carried out in a simple manner and in which uniform products are obtained in high yield. A process of this type should also be clearly superior to those known hitherto in economic and environmental terms.

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The invention therefore has the object of providing a process for the preparation of (metal) salts of alkylphosphonous acids which avoids the abovementioned disadvantages and with which the desired end products
25 can be prepared without difficulty.

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This object is achieved by a process of the type described at the outset, which comprises reacting elemental yellow phosphorus with halogen-free alkylating agents in the presence of at least one base.

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The reaction is preferably carried out in a two-phase system comprising at least one base and an organic solvent.

10 The alkylating agents are preferably dialkyl sulfates, trialkyl phosphates, dialkyl carbonates and/or formic acid ortho-esters. Particular preference is given to dimethyl sulfate.

15 The organic solvents employed are preferably straight-chain or branched alkanes, alkyl-substituted aromatic solvents, water-immiscible or only partially water-miscible alcohols or ethers, alone or in combination with one another.

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The organic solvent employed is particularly preferably toluene, alone or in combination with alcohols.

The bases are preferably hydroxides, carbonates, bicarbonates, amides, alkoxides and/or amine bases.

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Particular preference is given to the aqueous alkali metal hydroxides.

The reaction is preferably carried out in the presence
5 of a phase-transfer catalyst.

The phase-transfer catalyst is preferably a tetraalkylphosphonium halide, triphenylalkylphosphonium halide or tetraorganylammonium halide.

10

The temperature in the reaction is preferably from -20 to +60°C.

The temperature is particularly preferably from 0 to
15 30°C.

The reaction is preferably carried out under a pressure of from 0 to 10 bar.

20 The process according to the invention is preferably carried out by suspending or dissolving the yellow phosphorus in a solvent or solvent mixture and then reacting it with a halogen-free alkylating agent and at least one base.

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The yellow phosphorus and the halogen-free alkylating agent are preferably reacted with one another in a molar ratio of from 1:1 to 1:3, where the molar ratio of yellow phosphorus to base is from 1:1 to 1:5.

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The two-phase system obtained after the reaction is preferably separated and processed further as an aqueous phase.

10 The invention also relates to the use of the (metal) salts of alkylphosphonous acids prepared in accordance with the invention as precursors for chemical syntheses.

15 The invention also relates to the use of the (metal) salts of alkylphosphonous acids prepared in accordance with the invention for the preparation of organo-phosphorus compounds.

20 The invention also relates to the use of the (metal) salts of alkylphosphonous acids prepared in accordance with the invention as flame retardants or for the preparation of flame retardants.

25 The invention relates in particular to the use of the (metal) salts of alkylphosphonous acids prepared in

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accordance with the invention for the preparation of flame retardants for thermoplastic polymers, such as polyethylene terephthalate, polybutylene terephthalate or polyamide.

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The invention relates in particular to the use of the (metal) salts of alkylphosphonous acids prepared in accordance with the invention for the preparation of flame retardants for thermosetting resins, such as unsaturated polyester resins, epoxy resins, polyurethanes or acrylates.

Surprisingly, it has been found that elemental yellow phosphorus can be reacted with alkylating agents in a two-phase system (organic solvent/base) and, if desired, in the presence of a (phase-transfer) catalyst under extremely mild conditions by the process according to the invention to give the (metal) salts of the corresponding alkylphosphonous acid $RP(:O)HOH$.

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In addition, small amounts of dialkylphosphinic acids, trialkylphosphine oxide $R_3P(:O)$, dialkylphosphine oxide and unidentified phosphorus compounds may be formed; these can be removed from the product mixture in the usual manner. A further byproduct formed is hydrogen, which can easily be separated off from the reaction

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mixture. The abovementioned dialkylphosphinic acids can be separated off from the reaction mixture and employed or further processed elsewhere.

5 Surprisingly, the process according to the invention gives neither phosphine (PH_3) nor alkylphosphines (RPH_2 , R_2PH) in significant amounts. Through the choice of suitable reaction conditions - such as the addition of small amounts of alcohols to the organic phase - the
10 formation of all unidentified phosphorus-containing byproducts can be minimized to a surprisingly low content of a few mol% of the yellow phosphorus employed, in favor of the main product, the (metal) salts of alkylphosphonous acid.

15 The process according to the invention can be carried out, for example, by introducing the solvent together with the phase-transfer catalyst and warming the mixture to above the melting point of the yellow
20 phosphorus, then adding the elemental (yellow) phosphorus, cooling the mixture to temperatures of, for example, from -10 to $+30^\circ\text{C}$ with vigorous stirring, and subsequently adding the alkylating agent.

25 The reaction is initiated by addition of the base. When the reaction is complete, the reaction mixture can be

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diluted, for example with water, and the readily volatile components (H_2 , PH_3 , RPH_2 , R_2PH and excess alkylating agent, etc.) are subsequently removed.

- 5 This process gives a base-containing/organic two-phase system whose phases are separated. The contents from the phases are determined analytically.

10 The base-containing phase can be worked up by the known methods of the prior art in order to obtain the pure acids, such as, for example, alkylphosphonous acid (for example by ion exchange or distillation).

15 The reactants can also be combined in a different sequence, for example by introducing them continuously into a reactor (pressure tube, pressure reactor or cascade) in the above-defined molar ratio and removing them from the reactor again after a residence time of from 0.5 to 2 hours. The organic phase obtained after
20 the phase separation, which still contains the majority of any phase-transfer catalyst employed, is advantageously recycled.

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Example 1: Methylphosphonous acid

A solution of 52.2 g (0.1 mol) of tributylhexadecylphosphonium bromide in 1000 ml of toluene was introduced into a 5 l stainless-steel pressure reactor and pre-heated to 60°C. 62 g (2 mol) of yellow phosphorus were added, the mixture was cooled to -10°C with vigorous stirring, and 252 g (2 mol) of dimethyl sulfate were then metered in. The mixture was then warmed to 20°C, and 500 g of 40% aqueous sodium hydroxide solution were metered in over the course of 4 hours, during which the temperature was held at 20°C. Over the course of a further hour, 300 g of water were added, the mixture was then stirred for a further hour, and the reactor was subsequently decompressed via combustion. Two homogeneous liquid phases were obtained, which were separated and analyzed.

The aqueous phase (weight: 930 g) contained 54.6 mol% of methylphosphonous acid, 17.9 mol% of phosphorous acid, 19.7 mol% of hypophosphorous acid, 2.8 mol% of dimethylphosphinic acid in the form of their sodium salts and 3 mol% of dimethyldiphosphine.

The acids can be obtained from the salts prepared in Example 1 in a known manner, for example by ion exchange.

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Patent claims

1. A process for the preparation of (metal) salts of alkylphosphonous acids, which comprises reacting
5 elemental yellow phosphorus with halogen-free alkylating agents in the presence of at least one base.
2. A process as claimed in claim 1, wherein the
10 reaction is carried out in a two-phase system comprising at least one base and an organic solvent.
3. A process as claimed in claim 1 or 2, wherein the
15 alkylating agents are dialkyl sulfates, trialkyl phosphates, dialkyl carbonates and/or formic acid ortho-esters.
4. A process as claimed in one or more of claims 1 to
20 3, wherein the organic solvents employed are straight-chain or branched alkanes, alkyl-substituted aromatic solvents, water-immiscible or only partially water-miscible alcohols or ethers, alone or in combination with one another.

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5. A process as claimed in one or more of claims 1 to 4, wherein the organic solvent employed is toluene, alone or in combination with alcohols.
- 5 6. A process as claimed in one or more of claims 1 to 5, wherein the bases are hydroxides, carbonates, bicarbonates, amides, alkoxides and/or amine bases.
- 10 7. A process as claimed in one or more of claims 1 to 6, wherein the reaction is carried out in the presence of a phase-transfer catalyst.
- 15 8. A process as claimed in claim 7, wherein the phase-transfer catalyst is a tetraalkylphosphonium halide, triphenylalkylphosphonium halide or tetraorganylammonium halide.
- 20 9. A process as claimed in one or more of claims 1 to 8, wherein the temperature during the reaction is from -20 to +60°C.
10. A process as claimed in one or more of claims 1 to 9, wherein the temperature is from 0 to 30°C.

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11. A process as claimed in one or more of claims 1 to 10, wherein the reaction is carried out at a pressure of from 0 to 10 bar.
- 5 12. A process as claimed in one or more of claims 1 to 11, wherein the yellow phosphorus is suspended or dissolved in a solvent or solvent mixture and then reacted with a halogen-free alkylating agent and at least one base.
- 10 13. A process as claimed in one or more of claims 1 to 12, wherein the yellow phosphorus and the halogen-free alkylating agent are reacted in a molar ratio of from 1:1 to 1:3, where the molar ratio of
15 yellow phosphorus to base is from 1:1 to 1:5.
14. A process as claimed in one or more of claims 1 to 13, wherein the two-phase system obtained after the reaction is separated and further processed.
- 20 15. The use of a (metal) salt of an alkylphosphonous acid prepared by a process as claimed in claims 1 to 14 as a precursor for chemical syntheses.
- 25 16. The use of a (metal) salt of an alkylphosphonous acid prepared by a process as claimed in claims 1

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to 14 for the preparation of organophosphorus compounds.

17. The use of a (metal) salt of an alkylphosphonous
5 acid prepared by a process as claimed in claims 1
to 14 as a flame retardant or for the preparation
of flame retardants.

18. The use of a (metal) salt of an alkylphosphonous
10 acid prepared by a process as claimed in claims 1
to 14 for the preparation of flame retardants for
thermoplastic polymers, such as polyethylene
terephthalate, polybutylene terephthalate or
polyamide.

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19. The use of a (metal) salt of an alkylphosphonous
acid prepared by a process as claimed in claims 1
to 14 for the preparation of flame retardants for
thermosetting resins, such as unsaturated poly-
20 ester resins, epoxy resins, polyurethanes or
acrylates.

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Process for the preparation of (metal) salts of
- alkylphosphonous acids I

Abstract

The invention relates to a process for the preparation of (metal) salts of alkylphosphonous acids, which comprises reacting elemental yellow phosphorus with halogen-free alkylating agents in the presence of at least one base.

The invention also relates to the use of the (metal) salts of alkylphosphonous acids prepared by this process.